

Extension of the Kramers-Kronig method for polarized infrared reflectance spectra from the face of low-symmetry crystals

A.B.Kuz'menko, E.A.Tishchenko, A.S.Krechetov

P.L.Kapitza Institute for Physical Problems RAS,
Kosygina str., 2, Moscow, 117334, Russia

E-mail: abk@kapitza.ras.ru

Abstract

An extension of the Kramers-Kronig method for treatment of polarized infrared reflectance spectra from the face of low-symmetry crystals, where directions of principal dielectric axes depend on frequency, is proposed. It is shown, how to obtain the frequency dependencies of the complex reflectivity tensor components, using three reflectance spectra measured for different directions of linear polarization of the incident wave, when reflected wave is immediately sent to the detector. The problem is formulated in a form of the system of integral equations, and effective numerical technique is found for solving it. The question of the further recovery of the complex dielectric tensor on the base of the reflectivity tensor, is discussed. The case of the monoclinic crystals is considered in details. An example of the application of the extended Kramers-Kronig method to the reflectance spectra from the (ac)-face of the single-crystal bismuth oxide with the monoclinic lattice is given.

The Kramers-Kronig method (KK) along with the method of dispersionsal analysis (DA) is the most popular technique for treatment of reflectance spectra. As is known, it consists using the Kramers-Kronig integral transformations for recovery of frequency dependence of the phase θ of the complex reflectivity $r = \sqrt{R} \exp i\theta$ on the base of the real reflectivity R measured in wide enough frequency range. Applicability of such a procedure follows from the fact that the real part of the analytical function $\ln r = (1/2) \ln R + i\theta$, depends only on measured function $R(\omega)$, while its imaginary part is unknown function $\theta(\omega)$. So the function $\theta(\omega)$ may be found by integration:

$$\theta(\omega) = -\frac{\omega}{\pi} \int_0^\infty \frac{\ln R(\xi)}{\xi^2 - \omega^2} d\xi , \quad (1)$$

where integral with singularity must be treated in the sense of principal value ¹.

The next step is calculation of optical characteristics of matter. The normal incidence of electromagnetic wave to semi-infinite homogenous medium from vacuum is usually discussed. In this case the complex dielectric function ϵ may be calculated by formula:

$$\epsilon = \left(\frac{1-r}{1+r} \right)^2 , \quad (2)$$

that follows from the known Fresnel formulas.

An important advantage of the KK method (in comparison with DA) is that it allows to obtain the complex dielectric function without model calculations, provided that reflectance spectra are known in wide enough frequency range.

However, in the above form, the KK method is valid only when dielectric function of crystal and reflectivity coefficient can be considered as scalars. It is true when direction of electro-magnetic wave polarization coincides with one of the principal axes of the dielectric tensor $\hat{\epsilon}$. In this case eigenvalue of tensor $\hat{\epsilon}$ corresponding to particular axis, plays the role of scalar ϵ . This condition may be satisfied in entire spectral interval only for crystals with symmetry no lower than orthorombic. In the case of low symmetry crystals (with monoclinic or triclinic syngony) the dependence of direction of two or even three axes from frequency takes place (so called dispersion of dielectric axes). In monoclinic crystals only one axis is fixed in respect to crystallographic system of coordinates; two other rotate in the perpendicular plane. In triclinic crystals all axes have alternating direction. It doesn't allow to direct polarization of the incident radiation so that the vector of electric field and one of the principal axes of the dielectric tensor would be parallel. Hence the KK method (and the method of DA also) needs to be generalized.

Generalization of the method DA for phonon spectra in monoclinic crystals was first proposed by Belousov and Pavinich [3, 4], who introduced for each IR-active phonon an additional angular parameter which characterize direction of its dipole momentum and expressed tensor $\hat{\epsilon}$ through $4n + 3$ parameters, where n - number of phonon modes. For phonon parameters recovery several spectra, obtained for different polarizations of incident waves, were used. In [5] we applied this method in another form. It was shown that it is sufficient to measure three different spectra; every additional spectrum is their linear combination. We have measured spectra for three angles of polarization equidistant at 45° . Both in [3] and in [5] the reflectivity was calculated for the most popular situation when only one (the incident) wave is polarized. In this paper we offer method for the recovery of the complex reflectivity tensor starting from the same three spectra, as in the method DA in form [5]. This generalized KK method may be applied for any symmetry crystals and may be used in situations, when directions of principal axes of the dielectric tensor are not known in advance, as is in a case of low symmetry crystals.

¹Singularity may be eliminated by substitution of $\ln R(\xi)$ in numerator by $\ln R(\xi) - \ln R(\omega)$.

Calculation of the reflectivity tensor

On assumption of medium linear response, the normal reflection of electromagnetic wave from the crystal surface is wholly described by the complex reflectivity tensor:

$$\hat{r} = \begin{pmatrix} r_{xx} & r_{xz} \\ r_{zx} & r_{zz} \end{pmatrix}. \quad (3)$$

that associates complex amplitudes of incident (i) and reflected (r) waves: $\mathbf{E}_r = \hat{r}\mathbf{E}_i$ (for agreement with the notation in [3] and [5], we now assume that the wave vector of the incident wave is directed along the axis y). The components of the complex amplitude may be phase-shifted that corresponds to the elliptical polarization of wave. We will further assume that the medium is nongyrotropic (it means, for example, that no magnetic field is applied) and there is no spatial dispersion. In this case reflectivity tensor like dielectric tensor is symmetrical: $r_{xz} = r_{zx}$ [6, 7].

Having set the direction of the incident wave polarization by angle χ :

$$\mathbf{E}_i = E_i \begin{pmatrix} \cos \chi \\ \sin \chi \end{pmatrix}, \quad (4)$$

we will obtain for the measured reflectivity:

$$R(\chi) = \frac{|E_{rx}|^2 + |E_{rz}|^2}{|E_{ix}|^2 + |E_{iz}|^2} = |r_{xx} \cos \chi + r_{xz} \sin \chi|^2 + |r_{xz} \cos \chi + r_{zz} \sin \chi|^2. \quad (5)$$

The problem is to find spectral dependencies of six functions which determine the complex tensor \hat{r} using three reflectance spectra $R(0^\circ)$, $R(45^\circ)$ and $R(90^\circ)$. The structure of the right side of expression (5) doesn't allow to separate known and unknown functions by taking of logarithm as is possible in isotropic situation. Apparently, solving the problem of complex reflectivity tensor recovery by simple integration is impossible. Nevertheless, the problem may be formulated in terms of integral equations as is shown below.

Presence of two parts in (5) result from the fact that the reflected wave is generally elliptically polarized. If another polarizer (analyzer) were placed on the way of the wave, one of adding would be eliminated. But the scheme with only one polarizer is more preferable, because of the following reasons. Firstly, analyzer decreases intensity of light that falls onto detector and diminishes the signal-to-noise ratio. Secondly, the scheme discussed is the most typical for measurements of IR-spectra and often realized in commercial IR-spectrometers. At last, for the proposed variant of the method KK and for the method DA on monoclinic crystals [5] the same input spectra are used.

Three equations for unknown functions can be obtained if one associate them with the measured reflectance spectra by formula (5):

$$R(0^\circ) = |r_{xx}|^2 + |r_{xz}|^2, \quad (6)$$

$$2R(45^\circ) = |r_{xx} + r_{xz}|^2 + |r_{xz} + r_{zz}|^2, \quad (7)$$

$$R(90^\circ) = |r_{xz}|^2 + |r_{zz}|^2. \quad (8)$$

As was mentioned, additional equations for other polarization directions won't add any information, because every new spectrum must be a combination of first three ones. This statement follows from type of dependence of reflectivity from angle χ in accordance with (5).

Three more equations may be obtained from the KK equations for each component of the tensor \hat{r} :

$$\arg r_{xx}(\omega) = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln |r_{xx}(\xi)|}{\xi^2 - \omega^2} d\xi, \quad (9)$$

$$\arg r_{zz}(\omega) = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln |r_{zz}(\xi)|}{\xi^2 - \omega^2} d\xi, \quad (10)$$

$$\arg [1 + r_{xz}(\omega)] = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln |1 + r_{xz}(\xi)|}{\xi^2 - \omega^2} d\xi, \quad (11)$$

where all integrals are implied in the sense of principal value. Shift by one in the last equation is introduced because r_{xz} , unlike r_{xx} and r_{zz} , as function of the complex frequency may be equal to zero in the upper semiplane. It disturbs analiticity of function $\ln r_{xz}$ and results in inapplicability of KK transformations. In contrast, function $\ln(1 + r_{xz})$ is analytical in upper semiplane, because $|r_{xz}| < 1$.

Equations (6)-(8) along with (9)-(11) constitute complete system of equation, that may be solved numerically.

Calculation of the dielectric tensor

Having obtained frequency dependencies of all components of reflectance tensor, we must determine optical characteristics of crystal, particularly, the complex dielectric tensor $\hat{\epsilon}$.

Let us make general speculations, valid for all types of crystals, without any suggestion about orientation of dielectric axes. At first, let's remember that tensor $\hat{\epsilon}$ in the center of Brillouin zone (at $\mathbf{k} \rightarrow 0$) depends on direction of \mathbf{k} . It is governed by fact, that if wave polarization vector \mathbf{P} has longitudinal (relatively to \mathbf{k}) component \mathbf{P}_{\parallel} , then a macroscopic electric field appears inside crystal $\mathbf{E}_{\text{macro}} = -4\pi\mathbf{P}_{\parallel} = -4\pi\mathbf{k}(\mathbf{k}\mathbf{P})/|\mathbf{k}|^2$, which depend non-analytically from \mathbf{k} at $\mathbf{k} \rightarrow 0$ [8]. When the incident wave is normal to the surface of crystal the wave vector of induced polarization waves (polaritons) inside crystal will also be normal to the surface, because in accordance to boundary conditions the tangential component of \mathbf{k} must be constant. That's why all obtained values of tensor components will correspond to this direction of \mathbf{k} .

The knowledge of the reflectivity tensor \hat{r} with dimensions 2×2 , for one of the crystal faces is not enough to recover 3-dimensional tensor $\hat{\epsilon}$. Here we can obtain only partial information about $\hat{\epsilon}$. There is frequently used definition of the transverse reflectance tensor $\hat{\epsilon}_{\perp}$, that correlate the vector \mathbf{D} with the transverse component of vector \mathbf{E} : $\mathbf{D} = \hat{\epsilon}_{\perp} \mathbf{E}_{\perp}$. Because vector \mathbf{D} is always perpendicular to the surface, the tensor $\hat{\epsilon}_{\perp}$ is two-dimensional. In accordance to boundary conditions, the vector \mathbf{E}_{\perp} must be constant on both sides of surface. Hence, it is the tensor $\hat{\epsilon}_{\perp}$ that define reflectivity tensor \hat{r} . In [5] were derived formulas that show the relation among components of dielectric tensor and that of reflectance tensor. In the most compact form this relation may be expressed by formula:

$$\hat{\epsilon}_{\perp} = \left[(\hat{1} - \hat{r})(\hat{1} + \hat{r})^{-1} \right]^2, \quad (12)$$

that is similar to (2) ($\hat{1}$ is the unit tensor). Thus, in general case the KK method allows to recover tensor $\hat{\epsilon}_{\perp}$. The further analysis depends on the crystal symmetry and particular model, that define features of dielectric function. Relations between tensors $\hat{\epsilon}$ and $\hat{\epsilon}_{\perp}$ are described in details in [7].

Let us consider in details crystal with monoclinic lattice. In this case the IR-radiation reflectance from the monoclinic (ac)-plane doesn't reduce to the scalar case, because directions of two dielectric axes in this plane are frequency-dependent. Symmetry allows to extract from the tensor $\hat{\epsilon}$ the two-dimensional minor $\hat{\epsilon}_{ac}$, that determine dielectric properties in this plane. It coincides with the transverse tensor $\hat{\epsilon}_{\perp}$ and therefore is defined by formula (12). Only phonon modes B_u that have

dipole momentum in (ac)-plane contribute to $\hat{\epsilon}_{ac}$. In the model of polarized Lorenz oscillators this tensor equals:

$$\hat{\epsilon}_{ac}(\omega) = \hat{\epsilon}_{ac}^{\infty} + \sum_j \begin{pmatrix} \cos^2 \theta_j & \cos \theta_j \sin \theta_j \\ \cos \theta_j \sin \theta_j & \sin^2 \theta_j \end{pmatrix} \frac{S_j \omega_j^2}{\omega_j^2 - \omega^2 - i\gamma_j \omega}, \quad (13)$$

where S_j - oscillator strength, ω_j - transverse frequency, γ_j - damping coefficient, θ_j - angle between direction of dipole momentum and the axis x , $\hat{\epsilon}_{ac}^{\infty}$ - high frequency dielectric tensor. Summation is performed over all B_u -modes.

It is convenient to use $\epsilon_{ac} \equiv \text{Sp } \hat{\epsilon}_{ac} = \epsilon_{xx} + \epsilon_{zz}$ as uniform scalar characteristics for dielectric permeability in the (ac)-plane. In the model (13) it doesn't depend on directions of oscillator dipole momenta, in contrast to ϵ_{xx} and ϵ_{zz} apart:

$$\epsilon_{ac} = \text{Sp } \hat{\epsilon}_{ac}^{\infty} + \sum_j \frac{S_j \omega_j^2}{\omega_j^2 - \omega^2 - i\gamma_j \omega}. \quad (14)$$

Function (14) correspond to similar dependency of dielectric function from phonon mode characteristics in scalar case. Also, similar to a well-known rule for scalar case, transverse frequencies of modes B_u may be determined as positions of maxima on frequency dependency of $\text{Im } \epsilon_{ac}$. This rule, of course, is valid if modes doesn't strongly overlap (interval between two neighbour frequencies ω_j greater than their widths). Under this assumption the polarization angles θ_j (defined with ambiguity to π) may be approximately obtained from equation:

$$\cos 2\theta_j \approx \frac{\text{Im } \epsilon_{xx}(\omega_j) - \text{Im } \epsilon_{zz}(\omega_j)}{\text{Im } \epsilon_{xx}(\omega_j) + \text{Im } \epsilon_{zz}(\omega_j)}, \quad (15)$$

which might be drawn out if from imaginary part of sum (13) the contribution of only j -th mode (dominating at $\omega = \omega_j$) is considered. The choice of right root is determined by sign of off-diagonal element of $\text{Im } \epsilon_{xz}(\omega_j)$.

Features of numerical procedure

Having substituted:

$$ae^{i\alpha} = r_{xx}, \quad be^{i\beta} = r_{zz}, \quad ce^{i\gamma} = 1 + r_{xz}, \quad (16)$$

$$A = R(0^\circ), \quad B = R(90^\circ), \quad C = R(45^\circ) - (A + B)/2, \quad (17)$$

we'll find that system of equations (6)-(11) changes to:

$$A = a^2 + c^2 - 2 \cos \gamma + 1, \quad (18)$$

$$B = b^2 + c^2 - 2 \cos \gamma + 1, \quad (19)$$

$$C = ac \cos(\alpha - \gamma) + bc \cos(\beta - \gamma) - a \cos \alpha - b \cos \beta, \quad (20)$$

$$\alpha(\omega) = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln a(\xi)}{\xi^2 - \omega^2} d\xi, \quad (21)$$

$$\beta(\omega) = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln b(\xi)}{\xi^2 - \omega^2} d\xi, \quad (22)$$

$$\gamma(\omega) = -\frac{2\omega}{\pi} \int_0^\infty \frac{\ln c(\xi)}{\xi^2 - \omega^2} d\xi \quad (23)$$

with six unknown functions $a, b, c, \alpha, \beta, \gamma$ and three known functions A, B, C .

Let's vary unknown functions so that they satisfy system of equations. Inside measured frequency interval we choose quite dense grid of base points $\omega_1 < \omega_2 < \dots < \omega_n$. We approximately specify functions a, b, c by their values $a_i = a(\omega_i)$, $b_i = b(\omega_i)$ and $c_i = c(\omega_i)$, which assume as unknown variables. Then we calculate values of functions α, β, γ in base points, approximately substituting in formulas (21)-(23) integration by summation:

$$\alpha_i = \sum_{j=1}^n \sigma_{ij} \ln a_j, \quad \beta_i = \sum_{j=1}^n \sigma_{ij} \ln b_j, \quad \gamma_i = \sum_{j=1}^n \sigma_{ij} \ln c_j, \quad i = 1, \dots, n, \quad (24)$$

where coefficients σ_{ij} are expressed from $\omega_1, \dots, \omega_n$ according to method of approximate integration (type of interpolation function, method of extrapolation outside interval). For example, if one substitute a partly-constant function instead of real function $f(\omega)$:

$$f^*(\omega) = \begin{cases} f_1 & , \omega < (\omega_1 + \omega_2)/2 \\ f_j & , (\omega_{j-1} + \omega_j)/2 < \omega < (\omega_j + \omega_{j+1})/2, \quad j = 2, \dots, n-1 \\ f_n & , \omega > (\omega_{n-1} + \omega_n)/2 \end{cases}, \quad (25)$$

the following values of coefficients σ_{ij} will be obtained:

$$\sigma_{ij} = -\frac{1}{\pi} \cdot \begin{cases} \ln \frac{|2\omega_i - \omega_j - \omega_{j+1}|}{2\omega_i + \omega_j + \omega_{j+1}} & , j = 1 \\ \ln \frac{|2\omega_i - \omega_j - \omega_{j+1}|}{2\omega_i + \omega_j + \omega_{j+1}} - \ln \frac{|2\omega_i - \omega_{j-1} - \omega_j|}{2\omega_i + \omega_{j-1} + \omega_j} & , j = 2, \dots, n-1 \\ -\ln \frac{|2\omega_i - \omega_{j-1} - \omega_j|}{2\omega_i + \omega_{j-1} + \omega_j} & , j = n \end{cases}. \quad (26)$$

It corresponds to extrapolation of low- and high-frequency spectra by constant function, that is acceptable for dielectrics. This method was used to treating model and real spectra presented in this paper.

In each point there are three measured quantities $A_i = A(\omega_i)$, $B_i = B(\omega_i)$ and $C_i = C(\omega_i)$. After definition of set of values a_i, b_i and c_i and calculating with help of (24) α_i, β_i and γ_i , the following functional can be composed:

$$F = \frac{1}{3n} \sum_{i=1}^n \left[\left(\frac{A_i - A'_i}{\delta A_i} \right)^2 + \left(\frac{B_i - B'_i}{\delta B_i} \right)^2 + \left(\frac{C_i - C'_i}{\delta C_i} \right)^2 \right], \quad (27)$$

where trial quantities A'_i, B'_i and C'_i are expressed through $a_i, b_i, c_i, \alpha_i, \beta_i, \gamma_i$ in accordance with (18)-(20), and $\delta A_i, \delta B_i, \delta C_i$ are meansquare noise deviations of A_i, B_i, C_i .

Let's vary $3n$ quantities a_i, b_i and c_i to find a minimum of functional (27) as function of these quantities. As initial approximation it is reasonable to take $a_i = \sqrt{A_i}$, $b_i = \sqrt{B_i}$, $c_i = 1$, what is exact decision in a case when the reflectivity tensor (3) is diagonal. Set of values that makes functional approximatively equal one may be considered as approximate decision.

For functional minimization we have used the Marquardt-Levenberg nonlinear optimization method described in [9]. Partial derivatives of functional by variables a_i, b_i and c_i were calculated in analytical form. It is advisable to vary the density of base points according to density of spectrum "features" for diminishing n . In our calculations maximum number of base points n was 450. Despite great number of variables, minimization process converged quickly enough (5-7 iterations).

Certainly, it is not so obvious, why must minimization process always lead to true result, because functional F in multi-dimensional space may have local minima that do not correspond to required solution. Besides, the noise in input spectra may cause instability of minimization process. However

our experience shows that minimum which corresponds to right decision, is usually so deep that minimization procedure converges just to it. Experimental noise (in reasonable limits) doesn't affect the final result very much. Nevertheless, the convergence of method and its stability to experimental noises require special attention.

Application examples

Before application of the discussed method KK to real experimental data, we tested it on a set of model spectra obtained with a help of the dispersion formulas for monoclinic lattices [5], taking different sets of parameters of B_u phonon modes (frequencies, intensities, widths and polarization directions). To examine stability of the KK method to noise, additional noise with amplitude 5% was applied to initial spectra. Calculated by the KK method spectra for all components of the reflectivity tensor and the dielectric tensor were in a good agreement with that obtained directly by DA formulas. On the figure 1 results of one such test are showed.

Application of the discussed method to real reflectance spectra of single crystal α -Bi₂O₃, that has monoclinic lattice is shown as an illustration. On the figure 2(a) three reflectance spectra from the face (ac) are presented. They were used as input data for the generalized KK method. Details of experiment and results of data treatment by the DA method are described in [5].

Calculated by the KK method imaginary part of dielectric function ϵ_{ac} of bismuth oxide is presented on the figure 2(b) along with its frequency dependence calculated by the DA method. In general, a good match between these data may be noted. Several deviations, however, demonstrate that common description of dielectric function by Lorentz oscillators, isn't quite adequate. Inasmuch as discussion of optic properties of bismuth oxide isn't topic of this paper, we restrict ourselves by this notice.

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Figure captures

Fig.1. Results of the generalized KK method application to the model problem about reflection of IR-radiation from the (ac)-face of monoclinic crystal with two polarized in the (ac)-plane Lorentz oscillators, with characteristics: $\omega_1 = 200^{-1}$, $S_1 = 0.56$, $\gamma_1 = 10^{-1}$, $\theta_1 = 30^\circ$; $\omega_2 = 450^{-1}$, $S_2 = 0.79$, $\gamma_2 = 20^{-1}$, $\theta_2 = 60^\circ$. Components $\hat{\epsilon}_{ac}^\infty$: $\epsilon_{xx} = \epsilon_{zz} = 5.0$, $\epsilon_{xz} = 0.0$.

(a) Reflectance spectra at different polarization angles χ , obtained according to formulas of dispersional analysis with added artificial noise 5%. 1 - 0° (shifted at 1), 2 - 45° (shifted at 0.5), 3 - 90° .

(b) Components of reflectance tensor \hat{r} , obtained as a results of treatment spectra shown on figure 1a with help of KK method (dots) and directly with help of the DA formulas DA without additional noise (solid line). 1 - $\text{Im } r_{xx}$, 2 - $\text{Re } r_{xx}$, 3 - $\text{Im } r_{zz}$ (shifted at -1), 4 - $\text{Re } r_{zz}$ (shifted at -1), 5 - $\text{Im } r_{xz}$ (shifted at -2), 6 - $\text{Re } r_{xz}$ (shifted at -2.5).

(c) The imaginary part of the dielectric function ϵ_{ac} , calculated on the base of reflectivity tensor (figure 2a). Dots - reflectivity tensor was recovered using the KK method. Solid line - "true" reflectivity tensor calculated directly by the DA formulas.

Fig.2.

(a) IR reflectance spectra at $T=300$ K from the (ac) face of monoclinic crystal $\alpha\text{-Bi}_2\text{O}_3$ at different polarization angles χ . 1 - 0° (shifted at 2), 2 - 45° (shifted at 1), 3 - 90° .

(b) Frequency dependence $\text{Im } \epsilon_{ac}$, recovered by two methods from initial spectra drawn at figure 2a. Dots - function values obtained with a help of the generalized KK method. Solid line - results obtained with a help of the DA method [5].